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PATENT ABSTRACTS OF JAPAN

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(54) HIGHLY HEAT-RESISTANT POLYPROPYLENE FIBER

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a polypropylene fibrous material which does not melt at $\geq 170^{\circ}\text{C}$ autoclave curing temperature, especially polypropylene fiber or yarn excellent in shape retaining property in autoclave curing at $175\text{--}180^{\circ}\text{C}$ as fibrous material for reinforcing cement.

SOLUTION: This highly heat-resistant polypropylene fiber or yarn is obtained by melt-molding a polypropylene resin composition obtained by adding 0.001-5 pts.wt. nucleating agent forming β crystal to 100 pts.wt. homopolypropylene having $\geq 96\%$ isotactic pentad fraction and satisfying 0.3-30 g/10 min melt flow rate and drawing the molded material. In the fiber or yarn, the fiber shape is retained even in autoclave curing at 180°C .

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CLAIMS

[Claim(s)]

[Claim 1] The high thermal-resistance polypropylene fiber or yarn which comes to extend the polypropylene resin constituent which carried out 0.001-5 weight section addition of the ***** which makes a beta crystal form in the gay polypropylene 100 weight section with which an isotactic pentad fraction is 96% or more, and a melt flow rate fills 0.3-30g / 10 minutes after melting fabrication.

[Claim 2] High thermal-resistance polypropylene according to claim 1 or yarn used as fiber for cement reinforcement.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] Especially this invention relates to the fiber for cement reinforcement, or yarn about a high thermal-resistance polypropylene fiber or yarn.

[0002]

[Description of the Prior Art] As fiber for cement reinforcement, although asbestos was used conventionally, since asbestos has a bad influence on a human body, the use is regulated, and polyolefine fiber is beginning to be used as this alternative in recent years. In the stage of performing the fabrication, cement needs care-of-health process. 170-180 degrees C of care of health are performed within an autoclave (10 kgf/cm²) for dozens hours. However, in the usual polypropylene fiber, since the melting point is 160-165 degrees C, bearing this care of health cannot be finished, it dissolves, and the problem to which polypropylene does not exist as fiber in concrete after a care-of-health end arises. If the curing temperature of concrete is lowered to 165-170 degrees C, although a himself can be recuperated without a polypropylene fiber fusing, now, a productivity fall is caused to care of health by the need for a long time.

[0003]

[Problem(s) to be Solved by the Invention] The purpose of this invention offers the polypropylene fiber-like object which is not dissolved at the autoclave-curing temperature of 170 degrees C or more, the new polypropylene fiber which is especially excellent in form maintenance nature at the time of 175-180-degree C autoclave curing, or yarn as a fibrous object for cement reinforcement from the above-mentioned viewpoint.

[0004]

[Means for Solving the Problem] As a result of inquiring wholeheartedly that the above-mentioned technical problem should be attained, by adding a beta crystal nucleating additive in polypropylene with specific stereoregularity, this invention persons found out that a high thermal-resistance polypropylene fiber was obtained, and completed this invention. That is, this invention is the high thermal-resistance polypropylene fiber or yarn which comes to extend the polypropylene resin constituent which carried out 0.001-5 weight section addition of the ***** which makes a beta crystal form in the gay polypropylene 100 weight section with which an isotactic pentad fraction is 96% or more, and a melt flow rate fills 0.3-30g / 10 minutes after melting fabrication.

[0005]

[Embodiments of the Invention]

1. The polypropylene resin in a polypropylene resin this invention is gay polypropylene with which the isotactic pentad fraction (henceforth IPF) which is the index of stereoregularity is 96% or more, and a melt flow rate (henceforth MFR) fills 0.3-30. The melting temperature of the polypropylene resin concerned and its moldings is low in IPF being less than 96%, and it is not desirable as cement reinforcing materials. Since the pressure of a dice outlet rises too much that MFR is less than 0.3 at the time of melting fabrication (mainly spinning of fiber), it is not desirable. Moreover, if MFR exceeds 30, the amount component of macromolecules in polypropylene decreases, the fiber or the yarn after extension has few orientation crystals, as a result, the melting temperature of fiber or yarn becomes low, and it is not desirable as cement reinforcing materials. Furthermore, as for the molecular weight distribution (M_w/M_n) of polypropylene, 3.5-12 are desirable, and especially 4-9

are desirable.

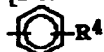
[0006] 2. As ***** in which the beta crystal used in a beta crystal nucleating additive this invention is made to form, they are a kind expressed with a general formula (1) or (2), or two sorts or more of amide system compounds.

R2-NHCO-R1-CONH-R3 (1)

R1 expresses the alicycle group dicarboxylic-acid residue of the saturation of the saturation of carbon numbers 1-24 or the aliphatic carboxylic-acid residue of an unsaturation, and carbon numbers 4-28, or an unsaturation, or the aromatic dicarboxylic-acid residue of carbon numbers 6-28 among [formula. It differs and R2 and R3 express the same or the basis shown by the cycloalkyl machine of carbon numbers 3-18, the cyclo alkenyl machine of carbon numbers 3-12, the general formula a, the general formula b, the general formula c, or the general formula d.]

[0007]

[Formula 1]

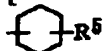


(a)

R4 expresses the alkenyl machine of the straight chain of carbon numbers 1-12, a branched-chain alkyl group or the shape of a straight chain of carbon numbers 2-12, and the letter of branching, the cycloalkyl machine of carbon numbers 6-10, or a phenyl group among [formula.]

[0008]

[Formula 2]

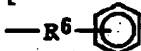


(b)

R5 is R4 and homonymy in a general formula a among [formula.]

[0009]

[Formula 3]

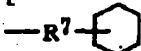


(c)

R6 expresses the alkylene machine of the shape of a straight chain of carbon numbers 1-4, and the letter of branching among [formula.]

[0010]

[Formula 4]



(d)

R7 is R6 and homonymy in a general formula c among [formula.]

[0011]

R9-CONH-R8-NHCO-R10 (2)

R2 and R10 are homonymy with R3 in a general formula (1) among [formula, respectively.] [in / a general formula (1) / in R1 and R9] / in / a general formula (1) / in R8]

Specifically, it is N and N'-dicyclohexyl. - The 2, 6-naphthalene dicarboxy amide, N, and N'-dicyclohexyl terephthalamide, N, and N'-diphenyl hexane diamide, N, and N'-JISHIKURO hexane carbonyl-p-phenylene diamine, N, and N'-dibenzoyl -1, 4-diamino cyclohexane, etc. are mentioned, and it is sold as ENUIESUTA - (tradename) from New Japan Chemical Co., Ltd. as these beta crystal *****.

[0012] this invention -- setting -- the amount of the beta crystal nucleating additive used -- the polypropylene resin 100 weight section -- receiving -- 0.001 - 5 weight section -- it is 0.005 - 1 weight section preferably Under in the 0.001 weight section, even if it is hard to generate a beta crystal in non-extended thread or yarn and being contained exceeding 5 weight sections, the predominance of an effect is not accepted but causes the thread breakage etc. conversely at the time of spinning. The ***** concerned may be blended at the time of polypropylene resin manufacture, and may be added to the resin prepared separately.

[0013] 3. To other polypropylene resin extension fiber or yarn of an additive this invention, the well-known modifier for polyolefines can be used together suitably conventionally according to the

purpose of use. For example, they are an antioxidant, an ultraviolet ray absorbent, a light stabilizer, an organic carboxylic acid, an antistatic agent, a surfactant, a neutralizer, a dispersant, an epoxy stabilizer, a plasticizer, lubricant, an antimicrobial agent, a flame retarder, a bulking agent, a foaming agent, a foaming assistant, a cross linking agent, a bridge formation assistant, a pigment, etc. As an antioxidant, a phenol system antioxidant, the Lynn system antioxidant, a sulfur system antioxidant, an amine system antioxidant, and vitamins are mentioned. As a neutralizer which served as the dispersant, a metallic soap, hydrotalcites, a lithium aluminum compound hydroxide salt, a silicate, a metallic oxide, a metal hydroxide, etc. are mentioned. Moreover, in order to raise the fiber in the inside of cement, or the dispersibility of yarn, it is also effective to add hydrophilic polymer, such as a polyethylene glycol and a polyethylene oxide, within the limits of 0.1 - 20 weight section.

[0014] 4. Fabrication of a polypropylene fiber, or the manufacture the non-extended fiber of the manufacture method (1) the non-extended Plastic solid of yarn is melting fabrication, and, generally is performed by melting extrusion molding. For example, the yarn for extension is obtained by carrying out melting extrusion of the polypropylene through a spinneret by obtaining the filament for extension and extruding polypropylene through a flat die or a ring die. Moreover, the split yarn for extension is obtained by judging after tubular-blown-film fabrication. In order to raise the ductility of the non-extended polypropylene Plastic solid obtained by doing in this way using the beta crystal nucleating additive content polypropylene constituent of this invention, it is desirable to promote beta crystal-ization of the crystal system of a non-extended Plastic solid further. It is desirable to cool slowly comparatively for that purpose at the time of a non-extended filament or yarn fabrication. It is also an effective method to carry out heating keeping warm directly under a spinning nozzle as the technique of generation promotion of a beta crystal.

[0015] (2) Extension operation extension operation can be performed with multi-stage [one step or two steps or more of]. The range of extension temperature is 70-150 degrees C, and it performs a hot platen, far infrared rays, etc. as a heat source in oven. In the case of fiber, in the case of two to 7 times, and yarn, draw magnification is four to 14 times preferably two to 16 times 1.5 to 10 times.

[0016] (3) Heat treatment thus the obtained polypropylene fiber, or yarn can be heat-treated by request (under a constraint). Generally 140-170 degrees C of this heat treatment are preferably performed within the limits of 150-165 degrees C for 1 to 20 minutes for 0.5 to 30 minutes. Crystallization of the orientation crystal section advances with this heat treatment, and the further high-melting point-ization is brought about. When carrying out autoclave curing, in order to raise over 2 - 5 hours slowly to the purpose temperature of 170-180 degrees C, it is the same situation as having heat-treated to the polypropylene fiber as a result. For this reason, you may carry out to serve also as heat treatment at the time of autoclave curing.

[0017] 5. As cement applied as cement reinforcing materials, the fiber or the yarn of a cement this invention to which the fiber or the yarn of this invention is applied as cement reinforcing materials can usually mention special cement, such as non-hydraulic cement, such as hydraulic cements, such as Portland cement, a white portland cement, alumina cement, a pozzolanic cement, magnesia cement, and Pozzolan cement, plaster, and coal, and an acid resisting cement, etc., for example. Moreover, it can obtain by blending thermosetting water soluble resins, such as the aggregates, such as a pebble and sand, paraffin, a wax, and resol type phenol resin, various kinds of polymer emulsions, a hardening accelerator, a concrete retarder, a water reducing agent, etc. with a kind or two sorts or more in the cement mentioned above as a cement constituent using the above-mentioned cement inorganic material, such as a calcium carbonate, a magnesium hydroxide, or a titanium white, and if needed, for example. When stiffening this cement constituent, as for the mixing ratio of the cement at the time of adding water to a cement constituent, and water, and the so-called C/W ratio, it is desirable to consider as the range of 1-10. The amount of a C/W ratio of water increases too much or less in one, and if the intensity of a cement hardened material does not become high enough but it becomes large from 10, the fluidity of a cement constituent will get worse.

[0018] 6. Face using the fiber or the yarn of a using-as cement reinforcing materials-fiber or yarn of this invention use mode this invention as cement reinforcing materials, and the forms used with the configuration of fiber or yarn differ. When yarn is made into cement reinforcing materials, the cement reinforcing materials concerning this invention are fixed to the semi-hardening object of a cement constituent by the rock bolt etc. in the stage which the above-mentioned cement constituent

has not hardened completely, and the method of supplying a cement constituent further etc. is used. [0019] Moreover, what is necessary is to mix into the above-mentioned cement constituent and just to use, after cutting cement reinforcing materials fiber in length of about 3-30mm preferably, when fiber is made into cement reinforcing materials. If fiber length is longer than 30mm, it will be hard coming to distribute in a cement constituent uniformly, and when it becomes conversely shorter than 3mm, it may be able to stop in this case, being able to acquire sufficient reinforcement effect. Moreover, if many [too], it will be hard coming to be unable to acquire sufficient reinforcement effect, if there are too few amounts of fiber cement reinforcing materials, but to distribute cement reinforcing materials about the amount in which the cement reinforcing materials of fiber are made to mix, uniformly. Therefore, although the amount of mixing of cement reinforcing materials fiber is the range of 0.5 - 30 weight section to the cement constituent 100 weight section, it is 1 - 15 weight section more preferably.

[0020] 7. Various cement products are mentioned as a product obtained by applying to the above-mentioned cement constituent by making the fiber of this invention, the fiber of the cement-product this invention using yarn as cement reinforcing materials, or yarn into cement reinforcing materials. For example, buildings, such as roads, such as the underwater structures, such as a tetrapod, a bridge, and a tunnel, the structure for railroads and a building, a residence, and a wall surface, a revetment block, a tile, etc. can be mentioned.

[0021]

[Example] Below, an example explains this invention in detail. The examining method in an example is as follows.

- (1) IPF: Macromolecules and 6,925 (1973) were asked by ¹³C-NMR spectral method of a publication. That is, it asked for the peak molar fraction which is equivalent to isotactic combination from the peak which shows the propylene monomeric unit which continued five pieces in ¹³C-NMR spectrum. Attribution of a peak was performed to Macromolecules and 8,687 (1975) by the method of a publication.
- (2) MFR: JIS K It measured at 2.16kg of loads, and 230 degrees C by 7210.
- (3) DSC measurement : about sample (extension fiber or yarn) about 10mg, to 210 degrees C, the temperature up was carried out and it measured [part / for scanning temperature/of 10 degrees C] from the room temperature.
- (4) Molecular weight distribution : it measured in GPC.
- (5) Spinning nature : the case where or more 3 thread breakage in 30 was carried out for continuation 60 minutes at the time of spinning was made into x.
- (6) Evaluation of the highest draw magnification : in the extension process, it considered as the scale factor of the upper limit which the thread breakage by extension does not produce for 20 minutes.
- (7) The fiber form evaluation technique after autoclave curing : the concrete test piece after care of health was broken, and the fiber configuration which remains in the cross section estimated. That is, that in which ** and a half do not remain what remains what all remains in the state of fiber mostly in the state of fiber to O and the half grade, either was made into x.

[0022] For 96% and MFR, 2g / 10 minutes, and molecular weight distribution (Mw/Mn) are [example 1IPF] as an antioxidant to the gay polypropylene 100 weight section of 6. After having used calcium stearate for Ir1010 and Ir168 (product made from Ciba-Geigy), using 0.1 weight ***** and the super mixer for the 0.05 weight section and the beta crystal nucleating additive ENUJIE star (New Japan Chemical Co., Ltd. make) as the 0.05 weight section and a neutralizer respectively and blending, melting kneading was carried out with the screw speed of 230 degrees C and 75rpm with the extruding press machine of 50mmphi, and pellet-like polypropylene was obtained.

[0023] Melt spinning of this was carried out using the multifilament spinning machine with a gear pump (dice : 0.8mm phix30 holes) by part for 300m/in the spinning temperature of 290 degrees C, and winding speed, and about 20-denier non-extended thread was obtained. Subsequently, it extended under conditions with a part for feed speed 50m/, the feed roll temperature of 90 degrees C, 130 degrees C [of heater temperature of an extending point], and a draw roll temperature of 110 degrees C. The highest draw magnification is 5 times and obtained extension thread 4.5 times. After cutting the extension fiber obtained as mentioned above in length of 15mm, Portland cement

(Chichibu Onoda Cement make), No. 8 silica sand, and water were made to mix into the cement constituent which blended and becomes by the weight ratio so that it may be set to Portland cement:silica sand:water=100:100:60. in addition, the mixing ratio of a cement constituent and the above-mentioned fibrous cement reinforcing materials -- a weight ratio -- cement constituent: -- fibrous -- it was referred to as cement reinforcing materials =100:1

[0024] the cement-fibrous cement reinforcing materials mixture obtained as mentioned above -- the inside of a mold with a length of 80mm, a width of face [of 30mm], and a height of 20mm -- slushing -- the following low pressure steam curing -- subsequently autoclave curing was performed on the 1st on the 1st

Low pressure steam curing: The isothermal 3 - 5-hour regimen after recuperating oneself 2 - 5 hours ago at 23 degrees C and raising at 20 degrees C/hour in speed to 65 degrees C. It applies after that for 10 to 15 hours, and cools slowly to 23 degrees C.

Autoclave curing: Supply to an autoclave iron pot after unmolding. the 3 - 5-hour isothermal after applying for 3 to 6 hours and heating and pressurizing to 180 degrees C and ten atmospheric pressure -- isotonic Then, water is filled in the space of the outer wall of an iron pot, and it cools over 7 - 10 hours to it.

[0025] The dissolution peak of the fibrous cement reinforcing materials by DSC measurement itself and end temperature are 180 degrees C and 184 degrees C, respectively, and marked melting point elevation being seen compared with the former, consequently holding the fiber gestalt after autoclave curing in 180 degrees C was checked. A result is shown in Table 1.

[0026] Except having made example 2 - 4 beta-crystal nucleating additive ENUIE star addition into 0.005, 1, and 5 weight sections, like the example 1, fibrous cement reinforcing materials 4 to 4.5 times the draw magnification of this were obtained, and the test sample was obtained like the example 1. Consequently, it was checked that 176-180 degrees C, 180-184 degrees C, and marked melting point elevation are seen, respectively, and the dissolution peak by DSC measurement and end temperature hold the fiber gestalt after autoclave curing in 180 degrees C. A result is shown in Table 1.

[0027] Like the example 1, fibrous cement reinforcing materials 4.2 times the draw magnification of this were obtained, and example 5IPF obtained the test sample like the example 1, except that MFR used 96.3% and molecular weight distribution used the gay polypropylene of 12 for 2g / 10 minutes. Consequently, it was checked that 181 degrees C, 184 degrees C, and marked melting point elevation are seen, respectively, and the dissolution peak by DSC measurement and end temperature hold the fiber gestalt after autoclave curing in 180 degrees C. A result is shown in Table 1.

[0028] Like the example 1, fibrous cement reinforcing materials 5.5 times the draw magnification of this were obtained, and example 6IPF obtained the test sample like the example 1, except that MFR used 97.0% and molecular weight distribution used the gay polypropylene of 6.2 for 15g / 10 minutes. Consequently, it was checked that 177 degrees C, 181 degrees C, and marked melting point elevation are seen, respectively, and the dissolution peak by DSC measurement and end temperature hold the fiber gestalt after autoclave curing in 180 degrees C. A result is shown in Table 1.

[0029] Like the example 1, fibrous cement reinforcing materials 6 times the draw magnification of this were obtained, and example 7IPF obtained the test sample like the example 1, except that MFR used 97.3% and molecular weight distribution used the gay polypropylene of 4.5 for 30g / 10 minutes. Consequently, it was checked that 176 degrees C, 180 degrees C, and marked melting point elevation are seen, respectively, and the dissolution peak by DSC measurement and end temperature hold the fiber gestalt after autoclave curing in 180 degrees C. A result is shown in Table 1.

[0030] Like the example 1, fibrous cement reinforcing materials 4.5 times the draw magnification of this were obtained, and example of comparison 1IPF obtained the test sample like the example 1, except that MFR used 92.0% and molecular weight distribution used the gay polypropylene of 6 for 2g / 10 minutes. Consequently, the dissolution peak by DSC measurement and the end temperature of 160 degrees C, 165 degrees C, and the melting point were low respectively, and it was checked after autoclave curing in 180 degrees C that the fiber gestalt is not held. A result is shown in Table 1.

[0031] Like the example 1, fibrous cement reinforcing materials 6.5 times the draw magnification of this were obtained, and example of comparison 2IPF obtained the test sample like the example 1,

except that MFR used 96.8% and molecular weight distribution used the gay polypropylene of 6.2 for 100g / 10 minutes. Consequently, the dissolution peak by DSC measurement and the end temperature of 166 degrees C, 169 degrees C, and the melting point were low respectively, and it was checked after autoclave curing in 180 degrees C that the fiber gestalt is not held. A result is shown in Table 1.

[0032] Except having made an example of comparison 3 beta-crystal nucleating additive ENUIE star's addition into 8 weight sections, like the example 1, fibrous cement reinforcing materials 4.5 times the draw magnification of this were obtained, and the test sample was obtained like the example 1. Consequently, although the dissolution peak by DSC measurement and the end temperature of 180 degrees C, 184 degrees C, and the melting point were high respectively and it was checked that the fiber gestalt is held after autoclave curing in 180 degrees C, the problem occurred to the spinning nature that thread breakage occurs frequently at the time of spinning. A result is shown in Table 1.

[0033] Except having made an example of comparison 4 beta-crystal nucleating additive ENUIE star's addition into the 0.0007 weight section, like the example 1, fibrous cement reinforcing materials 3.2 times the draw magnification of this were obtained, and the test sample was obtained like the example 1. Consequently, the dissolution peak by DSC measurement and end temperature were 172 degrees C and 176 degrees C, respectively, and the retentivity of the fiber gestalt was poor after autoclave curing in 180 degrees C. A result is shown in Table 1.

[0034] Instead of the example of comparison 5 beta-crystal nucleating additive ENUIE star, like the example 1, fibrous cement reinforcing materials 3 times the draw magnification of this were obtained, and the test sample was obtained for sorbitol system ***** like the example 1 except **** for the 0.2 weight sections. Consequently, the dissolution peak by DSC measurement and end temperature were 171 degrees C and 176 degrees C, respectively, and the retentivity of the fiber gestalt was poor after autoclave curing in 180 degrees C. A result is shown in Table 1.

[0035] Except **** for the 0.2 weight sections, like the example 1, fibrous cement reinforcing materials 3 times the draw magnification of this were obtained, and the test sample was obtained for NA11 (Asahi Denka Kogyo K.K. make) which is the Lynn system ***** instead of an example of comparison 6 beta-crystal nucleating additive ENUIE star like the example 1. Consequently, the dissolution peak by DSC measurement and end temperature were 172 degrees C and 177 degrees C, respectively, and the maintenance nature of the fiber form was poor after autoclave curing in 180 degrees C. A result is shown in Table 1.

[0036] MFR used 97.0% for 15g / 10 minutes, molecular weight distribution used the gay polypropylene of 6.2, instead of the beta crystal nucleating additive ENUIE star, like the example 1, fibrous cement reinforcing materials 4 times the draw magnification of this were obtained, and example of comparison 7IPF obtained the test sample for sorbitol system ***** like the example 1 except **** for the 0.2 weight sections. Consequently, the dissolution peak by DSC measurement and the end temperature of 171 degrees C, and 175 and the melting point were low respectively, and it was checked after autoclave curing in 180 degrees C that the fiber form is not held. A result is shown in Table 1.

[0037] Example of comparison 8IPF is 97.1%. MFR used for 30g / 10 minutes, molecular weight distribution used the gay polypropylene of 4.5, except **** for the 0.2 weight sections, like the example 1, fibrous cement reinforcing materials 4.5 times the draw magnification of this were obtained, and the test sample was obtained for NA11 (Asahi Denka Kogyo K.K. make) which is the Lynn system ***** instead of a beta crystal nucleating additive ENUIE star like the example 1. Consequently, the dissolution peak by DSC measurement and the end temperature of 172 degrees C, 176 degrees C, and the melting point were low respectively, and it was checked after autoclave curing in 180 degrees C that the fiber form is not held. A result is shown in Table 1.

[0038]

[Table 1]

	IPF (%)	MFR (g/10分)	Mw / Mn	造核剤A (重量部)	造核剤B (重量部)	造核剤C (重量部)	紡糸性	最高延伸倍率	延伸倍率	熱処理	融解ピーク温度(℃)	融解終了温度(℃)	180℃、10気圧オートクレーブ養生時の繊維の形態保持性
実施例1	96	2	6	0.1	—	—	○	5	4.5	有	180	184	○
実施例2	98	2	6	0.005	—	—	○	4.5	4	有	178	180	○
実施例3	98	2	6	1	—	—	○	5	4.5	有	180	184	○
実施例4	98	2	6	5	—	—	○	5	4.5	有	180	184	○
実施例5	98.3	2	12	0.1	—	—	○	4.7	4.2	有	181	184	○
実施例6	97	15	6.2	0.1	—	—	○	6	5.5	有	177	181	○
実施例7	97.1	30	4.5	0.1	—	—	○	6.5	8	有	178	180	○
比較例1	92	2	6	0.1	—	—	○	5	4.5	有	180	165	×
比較例2	98.8	100	6.2	0.1	—	—	○	7	6.5	有	166	169	×
比較例3	96	2	6	8	—	—	×	5	4.5	有	180	184	○
比較例4	98	2	6	0.0007	—	—	○	3.7	3.2	有	172	176	△
比較例5	98	2	6	—	0.2	—	○	3.5	3	有	171	176	△
比較例6	96	2	6	—	—	0.2	○	3.5	3	有	172	177	△
比較例7	97	15	6.2	—	0.2	—	○	4.5	4	有	171	175	△
比較例8	97.1	30	4.5	—	—	0.2	○	5	4.5	有	172	176	△

熱処理を160℃にて5分間行った。

造核剤A：β晶造核剤（エヌジェスター）

造核剤B：ソルビトール系造核剤

造核剤C：リン系造核剤（NA 11）

[0039] After fabricating a tubular blown film by 230 degrees C of extrusion temperatures, and the blow up ratio 0.90 using the polypropylene of the shape of a pellet prepared in the example 8 example 1, it extended by judging this crosswise. Extension considered the hot platen of 135 degrees C, heat set (heat treatment) temperature of 150 degrees C, and rolling-up roll speed as a part for 100m/, adjusted delivery roll speed, and determined draw magnification. In this way, extension yarn was obtained 12 times by the 13 times as many highest draw magnification as this. The test sample was obtained like the example 1 by making the aforementioned extension yarn into cement reinforcing materials.

[0040] Consequently, the dissolution peak of the stretched tape by DSC measurement and the end temperature of 180 degrees C, 183 degrees C, and the melting point were high respectively, and it was checked after autoclave curing in 180 degrees C that the fiber form is held. A result is shown in Table 2.

[0041] Except having made example 9 - 11 beta-crystal nucleating additive ENUJIE star addition into 0.005, 1, and 5 weight sections, like the example 8, yarn-like cement reinforcing materials 12 to 12.5 times the draw magnification of this were obtained, and the test sample was obtained like the example 1. Consequently, it was checked that 177-180 degrees C, 181-183 degrees C, and marked melting point elevation are seen, respectively, and the dissolution peak of the reinforcing materials by DSC measurement and end temperature hold the gestalt after autoclave curing in 180 degrees C. A result is shown in Table 2.

[0042] Like the example 8, yarn-like cement reinforcing materials 13 times the draw magnification of this were obtained, and example 12IPF obtained the test sample like the example 1, except that MFR used 97.0% and molecular weight distribution used the gay polypropylene of 6.2 for 15g / 10 minutes. Consequently, it was checked that 177 degrees C, 181 degrees C, and a marked melting point rise are seen, respectively, and the dissolution peak by DSC measurement and end temperature hold the form after autoclave curing in 180 degrees C. A result is shown in Table 2.

[0043] Like the example 8, yarn-like cement reinforcing materials 12.5 times the draw magnification of this were obtained, and example of comparison 9IPF obtained the test sample like the example 1, except that MFR used 92.0% and molecular weight distribution used the gay polypropylene of 6 for 2g / 10 minutes. Consequently, the dissolution peak by DSC measurement and the end temperature of 162 degrees C, 167 degrees C, and the melting point were low respectively, and it was checked after autoclave curing in 180 degrees C that the fiber form is not held. A result is shown in Table 2.

could not do molecular weight distribution with the gay polypropylene of 6.2 for 100g / 10 minutes, and it was not able to evaluate. A result is shown in Table 2.

[0045] Except having made sorbitol system ***** into the 0.2 weight section instead of the example of comparison 11 ENUIE star, like the example 8, yarn-like cement reinforcing materials 8 times the draw magnification of this were obtained, and the test sample was obtained like the example 1. Consequently, the dissolution peak by DSC measurement and end temperature were 170 degrees C and 175 degrees C, respectively, and the retentivity of the gestalt was poor after autoclave curing in 180 degrees C. A result is shown in Table 2.

[0046] Except having made NA11 into the 0.2 weight section instead of the example of comparison 12 ENUIE star, like the example 8, yarn-like cement reinforcing materials 8 times the draw magnification of this were obtained, and the test sample was obtained like the example 1. Consequently, the dissolution peak by DSC measurement and end temperature were 172 degrees C and 176 degrees C, respectively, and the retentivity of the gestalt was poor after autoclave curing in 180 degrees C. A result is shown in Table 2.

[0047] MFR used 97.0% for 15g / 10 minutes, molecular weight distribution used the gay polypropylene of 6.2, except having made sorbitol system ***** into the 0.2 weight section instead of the ENUIE star, like the example 8, yarn-like cement reinforcing materials 8 times the draw magnification of this were obtained, and example of comparison 13IPF obtained the test sample like the example 1. Consequently, the dissolution peak by DSC measurement and the end temperature of 171 degrees C, 175 degrees C, and the melting point were low respectively, and it was checked after autoclave curing in 180 degrees C that the form is not held. A result is shown in Table 2.

[0048]

[Table 2]

	IPF (%)	MFR (g/10分)	Mw /Mn	造核剤A (重量部)	造核剤B (重量部)	造核剤C (重量部)	成形性	最高延伸倍率	延伸倍率	熱処理	融解ピーク温度(℃)	融解終了温度(℃)	180℃、10気圧オートクレーブ養生時の繊維の形態保持性
実施例8	96	2	6	0.1	—	—	○	13.4	12.5	有	180	183	○
実施例9	96	2	6	0.005	—	—	○	13	12	有	177	181	○
実施例10	96	2	6	1	—	—	○	13.5	12.5	有	180	183	○
実施例11	96	2	6	5	—	—	○	13.5	12.5	有	180	183	○
実施例12	97	15	6.2	0.1	—	—	○	14	13	有	177	181	○
比較例9	92	2	6	0.1	—	—	○	13.5	12.5	有	162	167	×
比較例10	96.8	100	6.2	0.1	—	—	×	—	—	—	—	—	—
比較例11	96	2	6	—	0.2	—	○	9	8	有	170	175	△
比較例12	96	2	6	—	—	0.2	○	9	8	有	172	176	△
比較例13	97	15	6.2	—	0.2	—	○	9	8	有	171	175	△

熱処理を150℃にて5分間行った。

造核剤A：β晶造核剤（エノジエスター）

造核剤B：ソルビトール系造核剤

造核剤C：リン系造核剤（NA 11）

[0049]

[Effect of the Invention] The polypropylene fiber or yarn of this invention is the high thermal-resistance fiber or the yarn obtained from the constituent which added the beta crystal nucleating additive in the polypropylene of high stereoregularity, and if it uses as cement reinforcing materials, the fiber gestalt is maintained under a severe regimen, and it can fully demonstrate the effect as cement reinforcing materials.